DEVELOPMENT AND PRODUCING OF BULK NANOSTRUCTURED REFRACTORY METALS USING EQUAL CHANNEL ANGULAR PRESSING AND THEREMOMECHANICAL TREATMENT

Final Technical Report

by

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Abstract

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The results of investigations on development and application of ECAP for microstructure refinement in W and Ta are presented. Microstructure of the processed billets has been studied by the methods of optical and transmission electron microscopy (TEM) and the X-ray structural analysis. The influence of the number of passes on W microstructure evolution during the process of ECAP has been investigated. It has been shown, that the nanostructured state with a mean grain size equal to 100-200 nm is formed through additional high pressure torsion (HPT) in ECAP-processed W and Ta. Fabrication of bulk W and Ta billets with UFG structure has a strong influence on mechanical behavior of processed metals.

1. Introduction

Recent investigations [1-3] have demonstrated that ultrafine-grained (UFG) structure in metal materials processed by severe plastic deformation (SPD) provides great potential for achieving of new extraordinary properties. Equal-channel angular pressing (ECAP) is one of successful and commercially promising SPD techniques for fabricating of bulk UFG billets from different metals and alloys [1-5].

At the same time ECAP possesses a whole set of different technical parameters such as the pressing route, temperature, rate and level of straining and etc., which determine the degree of the structure refinement, and as a result, the enhancement of mechanical properties. Therefore to achieve high level of strength properties it is necessary to optimize the routes and regimes of ECAP.

In frames of our previous projects, supported by the European Research Office of the U.S. Army, we demonstrated for the first time a successful microstructure refinement through the ECAP technique in such hard-to-deform and low-ductility materials, as tungsten (W) and its alloys. It was shown, that ECAP of W led to formation of an UFG structure (about 0.5 μm in size), and as a result to enhancing of strength properties. At the same time, an increase in the number of cycles of ECAP represents a great interest, since this could lead not only to a further increase in strength, but also to enhancing of W ductility, that has been already observed in some metals [6]. This is, undoubtedly, very important for practical application of nanostructured W.

In our previous projects we also showed the possibility of strong structure refinement of another refractory metal – tantalum (Ta), using ECAP. Strength in Ta increased 3 times as a result of 8 ECAP cycles. As it has been established in the recent work [7] for UFG Ti, further improvement of properties is possible by means of combining of ECAP with additional thermo-mechanical treatment. Application of this approach to refractory materials presented an interest. That is why development and application of the combined treatment was the other essential task of the current project.

2. Multipass ECAP of W for grain refinement

2.1. Modernization of the die-set for multipass ECAP

The current project is aimed at development and processing of bulk nanostructured W and Ta billets, using multipass ECAP and additional thermo-mechanical treatment. To achieve the project goals it was necessary to design and manufacture the modernized die-set for ECA pressing of W. This die-set allows safely conducting pressing with a large number of cycles under the conditions of elevated temperatures and forces.

The earlier conducted investigations of processing of UFG W billets revealed [8], that low deformability of W in a temperature range of 900-1200 ºC is an important problem for multipass ECAP. It is possible to solve this problem at the expense of formation of the stress state, close to hydrostatic compression in the deformation region. At the same time the level of compressive stress should be high enough. Such a scheme of the stress state promotes to an increase in deformability of low-ductility materials.

It is possible to realize such conditions by means of producing backpressure in the output channel, in particular at the expense of increasing of friction forces, and application of such a die-set design and instrumental materials, which provide enhanced workability at high loads and temperatures. This concept was used as a basis of work on designing and manufacturing of the die-set. The scheme for the ECAP die-set, developed in the current project, is presented in Fig. 1. The die-set has double bending, which supplied the maximal possible level of compressive stresses in the forming insert and which is an important condition for enhanced workability of the die-set. The elongated output channel allowed creating additional backpressure at the expense of an increase in the friction surface, and to raise hydrostatic pressure in the deformation region. Application of a special insert allowed to improve tribological properties, i.e. to decrease the friction coefficient in the vertical channel and on its turn to decrease punch

unit loads and to increase its durability. High strength of the insert provided the enhanced durability of the die-set. The complex of the performed work allowed increasing the level of hydrostatic pressure in the deformation region up to 1800-2000 MPa and providing high workability of the die-set under the conditions of enhanced loads and elevated strain temperatures. Fabrication of such a die-set allowed successfully carrying out the investigation of the ECAP process at 1000-1200 ^{o}C and with multiple pressing cycles.

Data on W deformational behavior in the initial state and in the process of ECAP is very important when choosing ECAP regimes. In this connection in frames of the current project mechanical properties of W during upsetting tests in the temperature range of 1000-1250 ˚C were investigated. Mechanical tests were conducted on a universal test machine, supplied with the vacuum electric resistance furnace with an initial speed 7.1x10-3 c-1. The flow stress-temperature dependences were plotted according to the test results.

It is seen from Fig. 2, that one may observe a linear dependence of the flow stress on the test temperature in frames of the investigated temperature range. The flow stress values in the initial state increase from 190 to 255 MPa alongside with a decrease in temperature from 1250 to 1000 ˚C. Flow stresses increase as a result of ECAP. Alongside with an increase in the number of ECAP cycles the temperature dependence of the flow stress increases. The influence of the number of cycles of the flow stress is coming down in the temperature interval 1200-1250 ˚C.

Thus, taking into account relatively low flow stress values of W at elevated temperatures, we have come to a conclusion, that we should start multipass ECAP at 1150-1200 ˚ C and continue it at 1000-1150 ˚C.

At the same time it is necessary to consider the increase in the flow stress alongside with enlarging of the number of ECAP passes at temperatures lower than 1150 °C.

2.2. *Optimizing of ECAP regimes for fabrication of UFG W*

Recent investigation have made it possible to establish [8,9] that among different ECAP parameters, which influence most strongly the processing of integral UFG W billets, the temperature and level of the accumulated strain are the main ones. In its turn, the level of the accumulated strain is a function of strain intensity, which is assigned during ECAP by the value of the channels' intersection angle and the number of cycles. Usually ECAP of ductile materials is performed with an angle of channels'

intersection equal to 90^o [1]. However, it has been found that ECAP with an intersection angle of 120^o and the number of passes equal to 8 [8] is optimal to fabricate integral UFG billets. Further enhancing of properties in W is possible, if we increase the number of ECAP passes, starting from 8. In this case it is important to make the right choice of the billet's heating temperature, which allows conducting multipass ECAP without failure.

In frames of the current work the investigation of the influence of the number of cycles has been made at a constant heating temperature of the billet. Experimental investigation of ECAP, when the heating temperature was decreased in a range of 1200-1000^{o}C after each 2nd cycle, was carried out in order to study the influence of the heating temperature.

Commercially pure W, ordered by the Customer and supplied by PLANSEE, Austria, served the material for investigation. The material represented rods 16 mm in diameter and 100 mm in length. Structural studies, carried out with the help of optical metallography, showed that the structure of as-delivered W consisted of grains elongated along the rod' axis, which were 60-80 μm in diameter and 150 μm long (Fig. 3). Well-developed polygonized substructure with an average size of about 5 μm is observed in the electron-microscopy patterns inside of the grain structure (Fig. 4).

ECAP was conducted with the use of the hydraulic press with the maximal force equal to 160 ton-force. Traverse speed was 6 mm/sec. Heating of the billet was made in the air electric resistance furnace. The billet was preheated up to 1200-1000 ^{o}C. At the same time the die-set for ECAP was heated up to 500 ^{o}C. In order to lessen oxidation during heating, as well as to decrease the friction coefficient and the heat loss, the tungsten billet was introduced into a cylindrical steel covering.

The first two ECAP cycles were conducted at the heating temperature, close to the starting temperature of recrystallization, 1200 ^{o}C. Such a preliminary treatment contributed to lessening of forces during the subsequent pressing at lowered temperatures.

Investigations of W deformability during ECAP made it possible to reveal that pressing at a constant heating temperature equal to 1150 ^{o}C allowed to process integral billets with 11 cycles. An increase in the number of cycles led to cracks' development and to destruction of billets. ECAP with a decrease in the heating temperature from 1200 to 1000 ^{o}C allowed processing integral billets after 6 cycles. The view of W billets, processed by multipass ECAP (8 cycles), is presented in Fig. 5.

2.3. Formation of UFG structure and variation of W hardness depending on the number of ECAP cycles

Investigation of W structure formation during ECAP was conducted using optical and electron microscopy, the detailed description of which are presented in the previous reports. As the express method to control mechanical properties of the billets, served microhardness measurements by Vikers' method under a load of 200 g during 15 sec. Hardness measurements were carried out in the central part of the billet in the longitudinal and cross sections.

The metallography analysis of structure patterns of W, deformed using different regimes made it possible to establish, that a great number of ECAP cycles at a constant heating temperature contributed to formation of homogeneous and uniformly refined structure, as compared with a lower number of ECAP cycles with a decrease in the heating temperature from 1200 to 1000 ^{o}C.

Fig. 6 and 7 presents W microstructure patterns obtained by ECAP according to the following regimes:

1) 2 cycles at the heating temperature of 1200 ^{o}C + 9 cycles at 1150 ^{o}C

2) 2 cycles at 1200 ^{o}C, 2 cycles at 1100 ^{o}C and 2 cycles at 1000^{o}C.

Two areas, which are characterized by etchability (Fig. 6 and 7) could be distinguished in W microstructure patterns. Microhardness (Hv) measurements showed that values Hv in bright structure areas are 300 units less, than in the dark ones. As a consequence, dark areas correspond to the structure, where a stronger microstructure refinement took place. As it is seen from Fig. 6 and 7, the region covered by bright structure areas of W, deformed according to the second ECAP regime is larger than in the billets, subjected to ECAP according to the first regime with a greater number of cycles.

Electron – microscopy investigations of dislocation structure allowed establishing that when deforming W according to the 2nd ECAP regime the average grain/subgrain size is equal to about 0.5 μm, and according to the first one it consisted about 1 μm (Fig. 8).

Hardness measurements showed, that when deforming W according to the first and the second ECAP regimes, the average microhardness values are rather similar and they increased from 5 GPa in the initial state to 5.7 GPa.

Thus, in order to meet the project goals, the decision was made to process a batch of W billets with a different number of ECAP cycles at 1150 ºC. Structure investigations

showed that alongside with an increase in the number of ECAP cycles, homogeneity and uniformity of refinement of W structure is increased.

Investigation of the influence of annealing on W structure and properties after 11 ECAP cycles at 1150 ºC showed that heating at 1150 ºC for 1 hour led to formation of separate assembly, consisted of regular-shaped grains, that testified to beginning of recrystallization processes development (Fig. 9 a) and as a result, to a decrease in microhardness value by 500 MPa. Structure variations, observed after annealing at 1000 ºC, are connected, most of all, with sharpening of grain boundaries, which is a characteristic feature of recovery processes development (Fig. 9 b). Thin boundary lines in metallographic patterns surrounds areas of a smaller size, having a homogeneous contrast. At the same time the microhardness value decreases by 300 MPa as compared with the ECAP state.

Fig. 10 and 11 presents W structure patterns, observed using the optical microscope, after 8 and 12 ECAP cycles, accordingly. With an increase in the number of passes from 8 to 12, the share of bright matrix areas reduces, that testifies to an increase in homogeneity and structure refinement.

Variation of W dislocation structure after 8 and 12 ECAP cycles is presented in Fig. 12. The size of subgrains reduces from 1 μm after 8 cycles to 0.5 μm after 12 cycles. At the same time microhardness value increased from 5.4 to 5.8 GPa.

3. Fabrication of UFG W with enhanced hardness

In order to fabricate UFG billets, possessing enhanced hardness characteristics, investigations of ECAP W, subjected high pressure torsion, as well as to several ECAP cycles at lowered temperatures, have been conducted in frames of the current project.

3.1. High pressure torsion of W

W samples were subjected to ECAP and upsetting in order to enhance to an extent deformability of the material before the subsequent high pressure torsion.

W billets in a shape of a disk 9 mm in diameter and 2 mm high were deformed by upsetting by 30% at 600 ^{o}C. In order to reduce oxidation and to suppress cracks' development, upsetting of W was made in the protecting covering. The samples after upsetting were subjected to HPT. HPT straining was performed using the anvils pre-heated up to 500 ^{O}C under an imposed pressure of about 4 GPa on specimens in the shape of a disc with electropolished surface. In order to process HPT W samples we

used anvils, one of which had a groove 1 mm deep and 11 mm in diameter. Application of this-type designed anvils allowed fabricating samples about 1 mm high and about 10 mm in diameter without cracks.

Microhardness measurements were made using the Vickers' method along the top-to-bottom diameter of the metallographically polished HPT disk under a load of 200 g applied for 15 sec. As can be seen in Fig. 13, HPT straining leads to an increase in the microhardness from 5.5 GPa up to 10 GPa. Investigations of structural macrohomogeneity by measurements of radial distribution of microhardness have shown that the microhardness of the top side disc sample is somewhat higher than in the bottom, and in the center of HPT samples it is somewhat lower than in the periphery, but in all cases it is higher than 7 GPa. Two W samples made with this method were delivered to Prof. Evan Ma from John Hopkins University for further studies of mechanical properties, as it was agreed with the Customer.

3.2. ECAP at lowered temperatures

The conducted investigations allowed establishing efficiency of ECAP at lowered temperatures to process billets with high hardness during a smaller number of cycles. However, reducing of the billets' deformability and their destruction even after 1-2 pressing cycles, as a result of which one may observe formation of heterogeneous structure, is a disadvantage of ECAP at lowered temperatures. In this connection, W billets subjected to 8 ECAP passes at 1150 ^{o}C served as billets for ECAP at low temperatures. At the same time ECAP was conducted using the die-set with an angle of channels' intersection equal to 135^{o}, which allowed obtaining integral W billets after 2 cycles at 1000-950 ^{o}C. Microhardness of UFG W in these billets consisted more 6 GPa.

4. The structure and properties of Ta after ECAP

In our previous projects we also showed the possibility of strong structure refinement of another refractory metal – tantalum (Ta), using ECAP. After 8 cycles of ECAP, carried out at room temperature, the strength of Ta increased 3 times. In the current report the results of further investigations of ECAP Ta, directed to study of influence of number of ECAP passes on its structure and properties, are presented.

4.1. The structure and properties of Ta, subjected to ECAP

The die-set for ECAP of Ta was developed and described in the previous project report. The given technology makes it possible to carry out multipass ECAP of Ta, using

the die-set with the angle of channels intersection 90^o at room temperature. To obtain integral billets the given die-set with backpressure was used in the current project. ECAP of Ta was conducted along Bc route (rotation of a billet about 90^o clockwise along the longitudinal axis of a billet). The given route is the most effective to form equiaxed UFG structure [1,2]. An integral Ta billet was obtained as a result of ECAP along the Bc route after 12 passes.

The structural investigations of ECAP Ta billets were carried out with the help of optical microscopy in the longitudinal and transverse directions. The samples for OM after preliminary mechanical and electrodepositing polishing were etched in NH4F+HF water solution.

The mechanical properties of ECAP billets were defined from tensile tests and measurement of microhardness. Microhardness was measured in cross and longitudinal sections of ECAP billet by Vikers' method under load of 200 g during 15 sec.

Mechanical tests were conducted on the universal dynamometer Instron 1185 at room temperature with the initial strain rate 5.5×10^{-4} s^{-1} on the plane samples with the sizes of the working part 3x1.5x15 mm. According to the tests data engineering stress-strain curves were plotted.

The microstructure of Ta in the initial state represents equiaxed grains with the size 30 μm in longitudinal and cross sections (Fig. 14). ECAP along the Bc route leads to considerable microstructure refinement, the character of which depends on the number of passes.

In Fig. 15 the microstructure of Ta observed in the optical microscope after 2 ECAP cycles is presented. As seen, after 2 ECAP cycles the initial grains take the elongated form and are oriented at a definite angle to the longitudinal axis of a billet. In the longitudinal section the grain size increased 2.5 times and decreased twice in the cross section.

ECAP after 8 passes leads to considerable refinement of grain structure and increase of microstructure homogeneity (Fig. 15c,d). Ta microstructure after 12 passes is also presented in Fig. 15e,f. As seen, after such treatment formation of high dispersion, more equiaxed microstructure in longitudinal and cross sections of a billet is observed.

In Fig. 16 stress-strain curves before and after ECAP with different number of passes are presented. The influence of the number of ECAP cycles on the mechanical properties of Ta is shown in Fig. 17. The mechanical tensile tests showed that as a result of ECAP growth of Ta strength and reduction of ductility took place. The strongest

change of mechanical properties is observed after the first two cycles. Further, monotonous increase of strength takes place. After 12 cycles of pressing ultimate tensile strength during pulling increased almost by 4 times as compared to the initial state. Meanwhile, as a result of drastic fall of uniform elongation the total elongation decreased 5 times.

The presented results of structural investigations and mechanical properties have shown that ECAP of Ta considerably increases material strength but decreases ductility. The problem of increasing the complex of mechanical properties of metals and alloys with nano and ultrafine grained structure is hard-to-perform task and requires development of special approaches. One of such approaches for pure metals is creation of UFG structure with non-equilibrium high-angle boundaries [10] that may be realized with the help of special additional thermo-mechanical treatment of ECAP samples.

Therefore in the current report the results of investigation of the influence of low temperature annealing on the structure and mechanical properties of Ta after ECAP are presented.

4.2. Influence of low-temperature annealing on the structure and properties of ECAP Ta

The dependence of hardness on the temperature of isochronic annealing of ECAP Ta after 12 cycles is presented in Fig.18. As it is seen from Fig. 18 with temperature rising the change of hardness is non-monotonous at maximum 400 ^{o}C.

The investigation of the ECAP Ta structure after annealing within the temperature interval of 200-600 ^{o}C, conducted with the help of optical microscopy, did not reveal recrystallization characteristics, associated with formation of new grains (Fig. 19). It is evident, that the observed decrease of ECAP Ta microhardness after annealing within the temperatures of 450-600^{o}C is the result of perfecting of dislocation structure. However, the reason for increase of hardness after annealing at 400 ^{o}C is vague so far.

As a result of isothermal annealing at 400 ^{o}C during 15 hours hardness of Ta increased from 2550 after 8 ECAP cycles to 4000MPa (Fig. 18). Mechanical tests showed that ultimate tensile strength of Ta increased from 730 MPa after 8 ECAP cycles to 1000 MPa after additional low-temperature annealing at 400 ^{o}C during 15 hours. Meanwhile, ductility of Ta practically did not change.

The observed change of mechanical properties of ECAP Ta as a result of low-temperature thermal treatment may be conditioned by influence of interstitial impurities such as carbon, oxygen or nitrogen [11] and requires further investigations.

4.3. High-strength state of Ta, obtained by ECAP + HPT

In the current report the first results of the investigation of influence of additional treatment by means of HPT on the properties of Ta after ECAP are presented. The investigation of mechanical properties was conducted by measuring values of microhardness using a load of 200 g during 15 sec. HPT of Ta after ECAP (12 passes) was carried out at room temperature under the applied pressure of 6 GPa and with 10 turns. As a result of HPT billets in the form of a disk 10 mm in diameter and 0.5 mm in thickness were obtained.

The sample diameter distribution of values of microhardness is shown in Fig. 20. As it is seen from Fig. 20, HPT of Ta billets leads to increase in microhardness value from 250 to 420 Hv.

Thus, the use of HPT after ECAP leads to essential additional rise of material strength, which is connected with further refinement of its microstructure till nanosizes.

Formation of nanostructured state in tantalum may cardinally change its mechanical behaviour (strain localization, ductility) and this problem demands further detailed investigations.

5. Conclusions

A batch a ECAP W billets after 4, 8, and 12 passes was processed in frames of the current project. It was established, that multi-pass ECAP of W at elevated temperatures contributed to fabrication of integral bulk billets possessing UFG structure with the size of grains less than 1 μm. ECAP at 1150 °C led to the increase in the microhardness value from 5 GPa in the initial state to 5.8 GPa after 12 cycles.

The conducted investigations showed the efficiency of ECAP of W at lowered temperatures in order to process billets with enhanced hardness. Performing of ECAP using the die-set with the angle of the channels' intersection equal to 135^{o} allowed to additionally increase microhardness above 6 GPa.

In order to fabricate UFG Ta billets, multipass ECAP was successfully conducted at room temperature. The ultimate tensile strength of Ta after 12 cycles increased practically four times. Investigations of application of additional treatment by means of high pressure torsion of ECAP W and Ta billets showed the potential for development of ECAP-based combined treatment methods in order to fabricate UFG materials, possessing high strength and ductility characteristics.

In this connection, perfecting of ECAP at lowered temperatures, as well as development and application of such methods, as hot hydrodynamic extrusion present an interest. A special scheme of the stress-state state during such treatment, which is similar to hydrostatic compression, will allow achieving high strain levels e=0,4-1 at 700-800 ^{o}C. Investigations of structure and properties of HPT-processed W nanostructured billets, having the shape of a disk 20 mm in diameter, is also very perspective.

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7. Appendix (please, see file N62558-04-M-0009_P3_Figures.pdf attached).

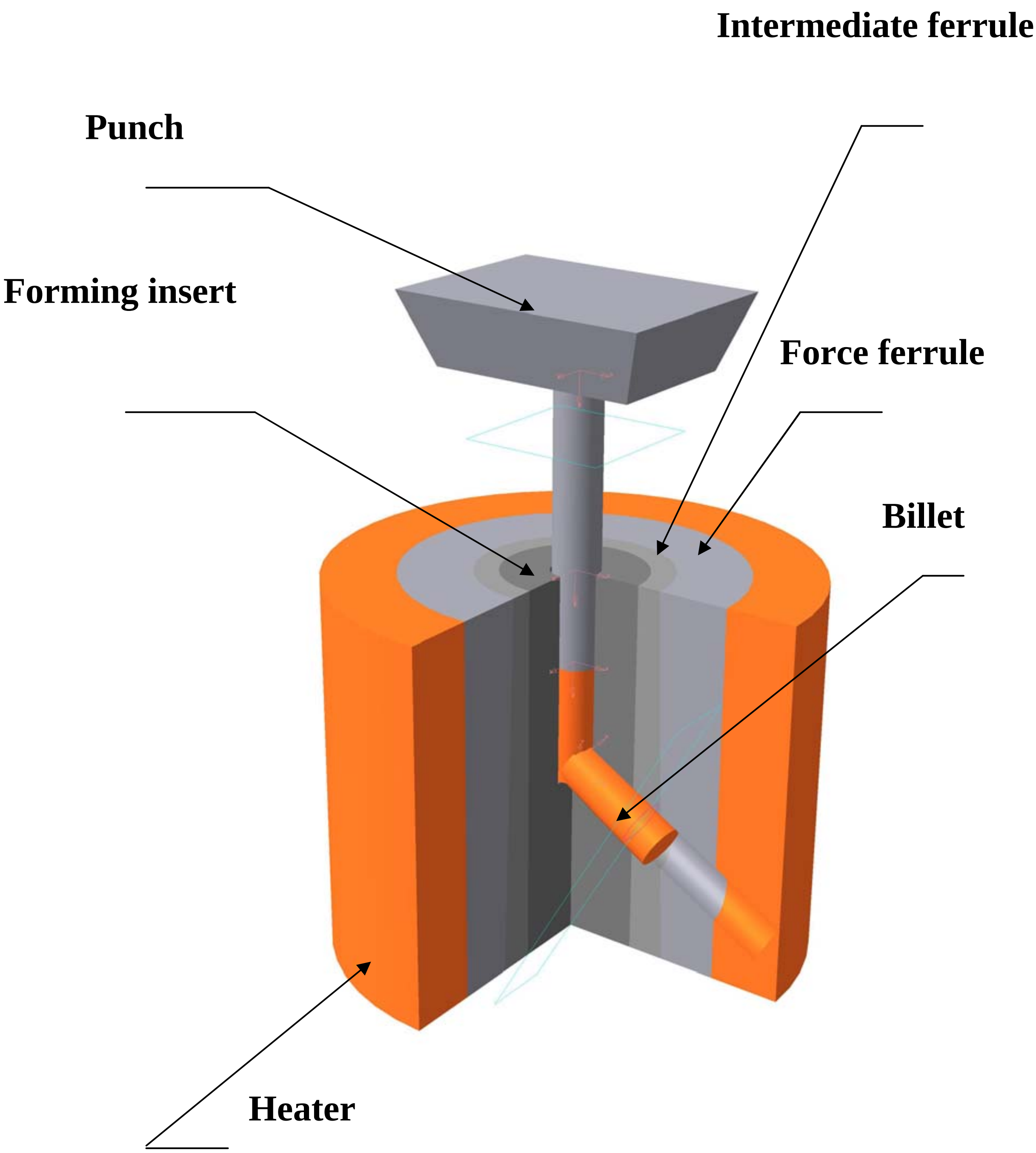


Fig. 1. Scheme of the modernized ECAP die-set

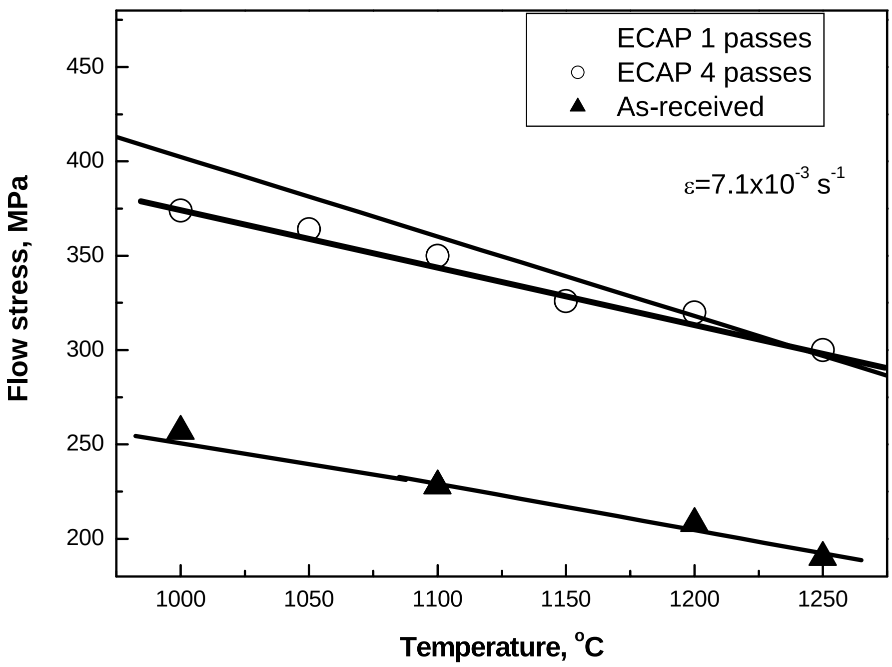


Fig.2. Influence of the number of ECAP passes on the temperature dependence of W flow stress during upsetting tests

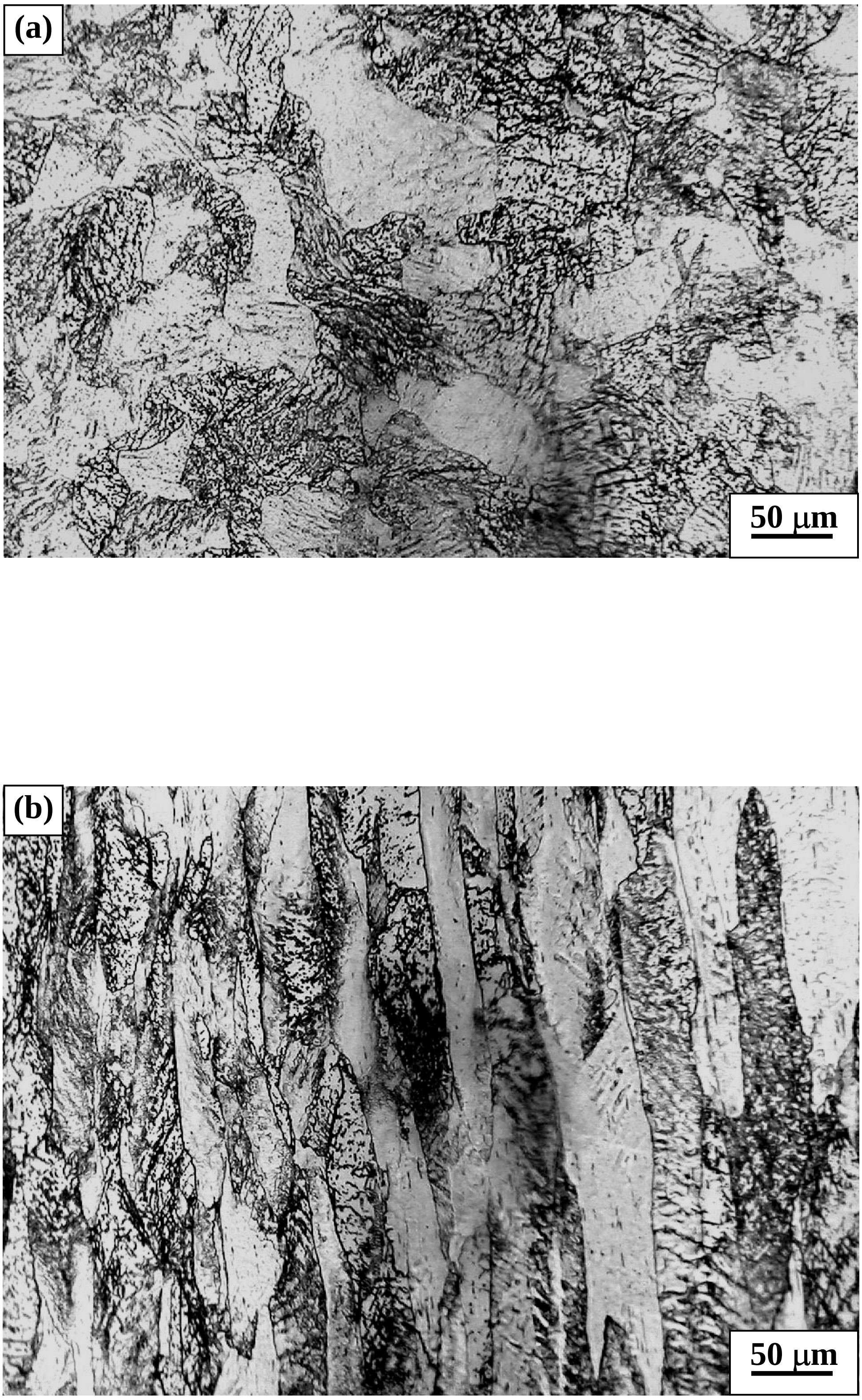


Fig. 3. W microstructure in the initial state:
a-cross section, b- longitudinal section

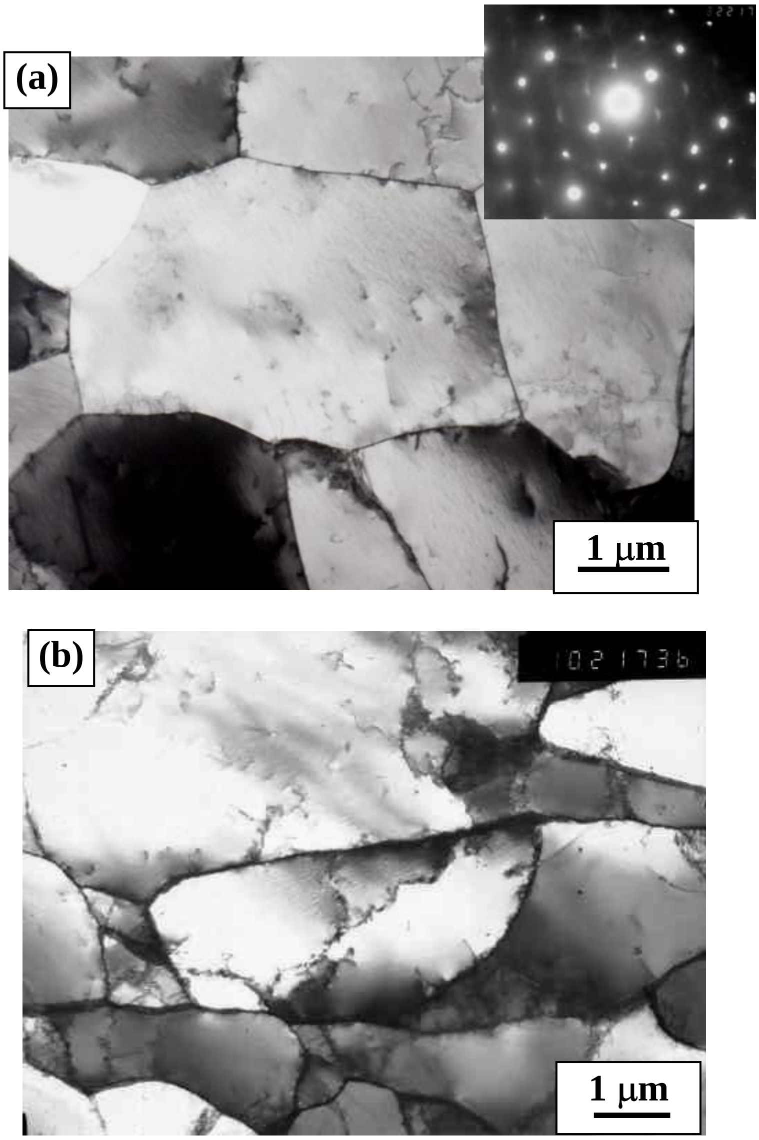


Fig 4. Electron-microscopic pattern of as-delivered W dislocation structure: a- cross section, b- longitudinal section

Fig. 5. W billets after 8 ECAP passes at 1150 ^{o}C

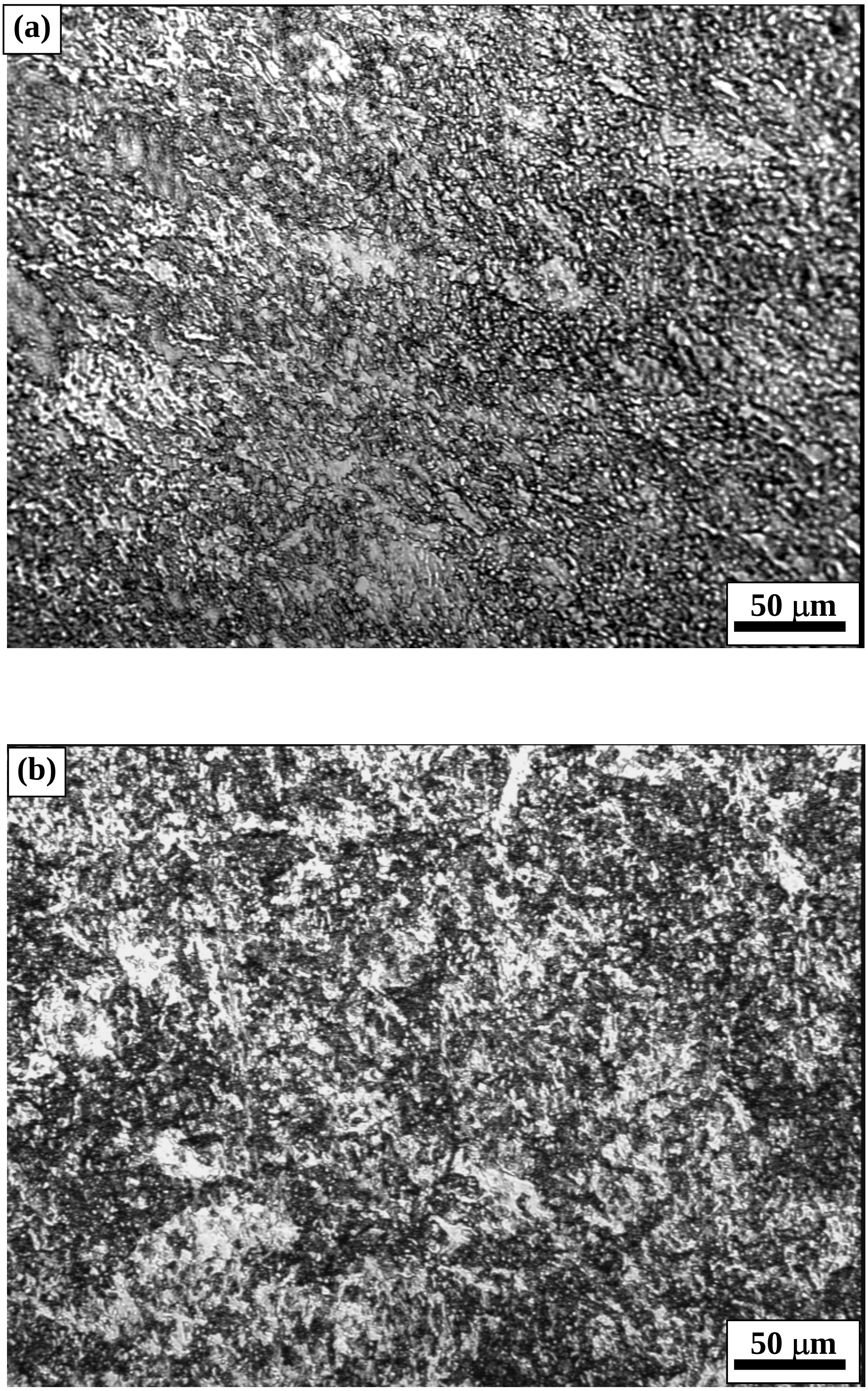


Fig. 6. Metallographic pattern of W after 11 ECAP cycles at 1150 °C: a- longitudinal section, b- cross section

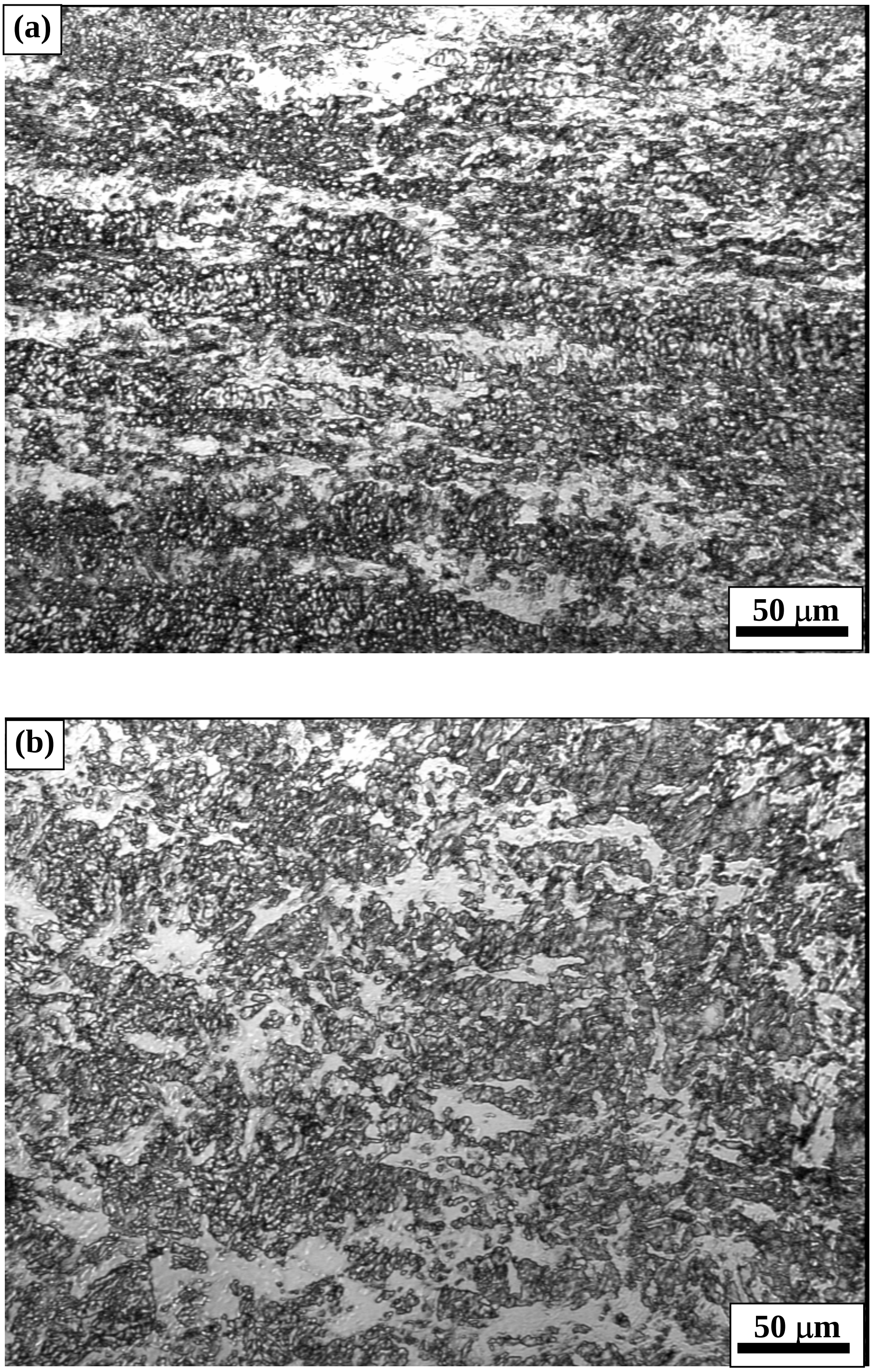


Fig. 7. Metallographic pattern of W after ECAP by regime:
2 cycles at 1200 °C +2 cycles at 1100 °C and 2 cycles at 1000 °C :
a- longitudinal, b- cross sections

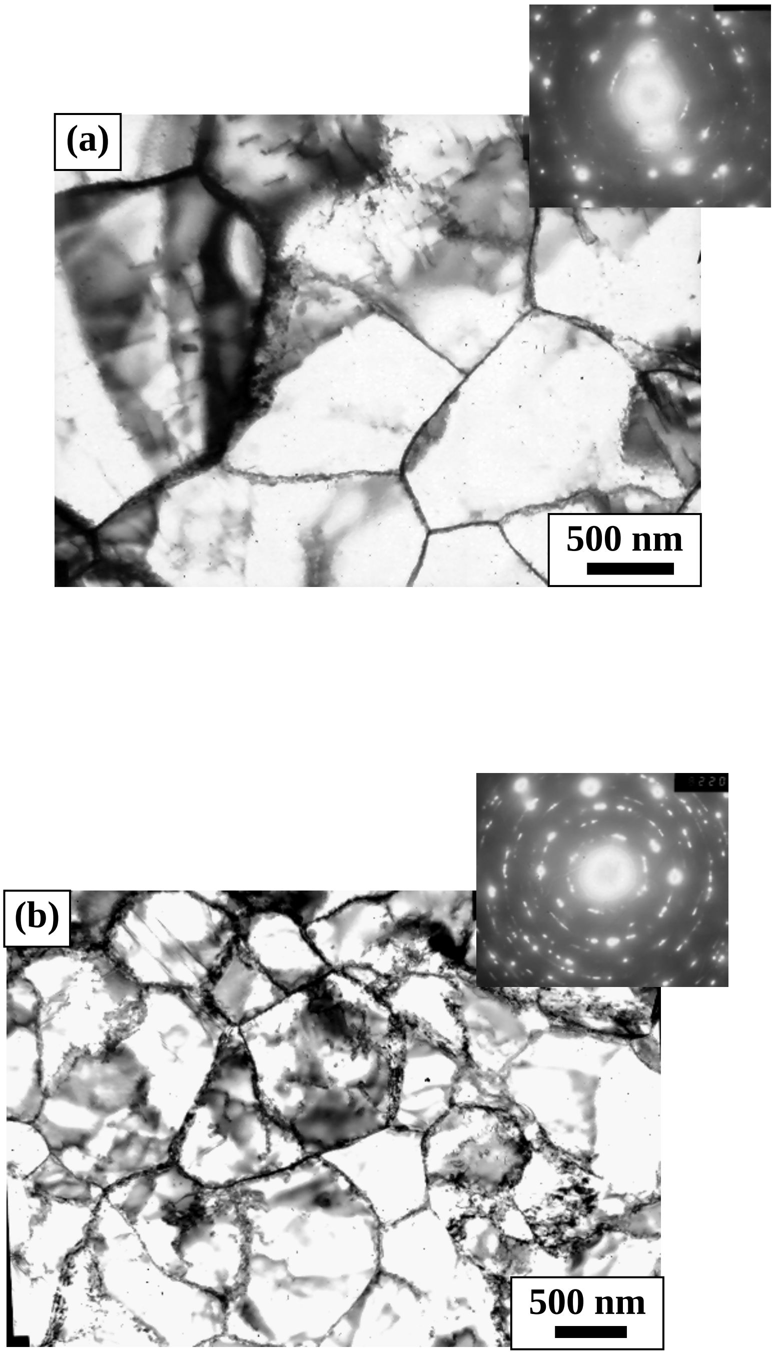


Fig. 8. Electron-microscopic patterns of W dislocation structure by regimes: (a)-1150 ^{o}C and 11 cycles; (b)-2 cycles at 1200 ^{o}C +2 cycles at 1100 ^{o}C and 2 cycles at 1000 ^{o}C. (cross section)

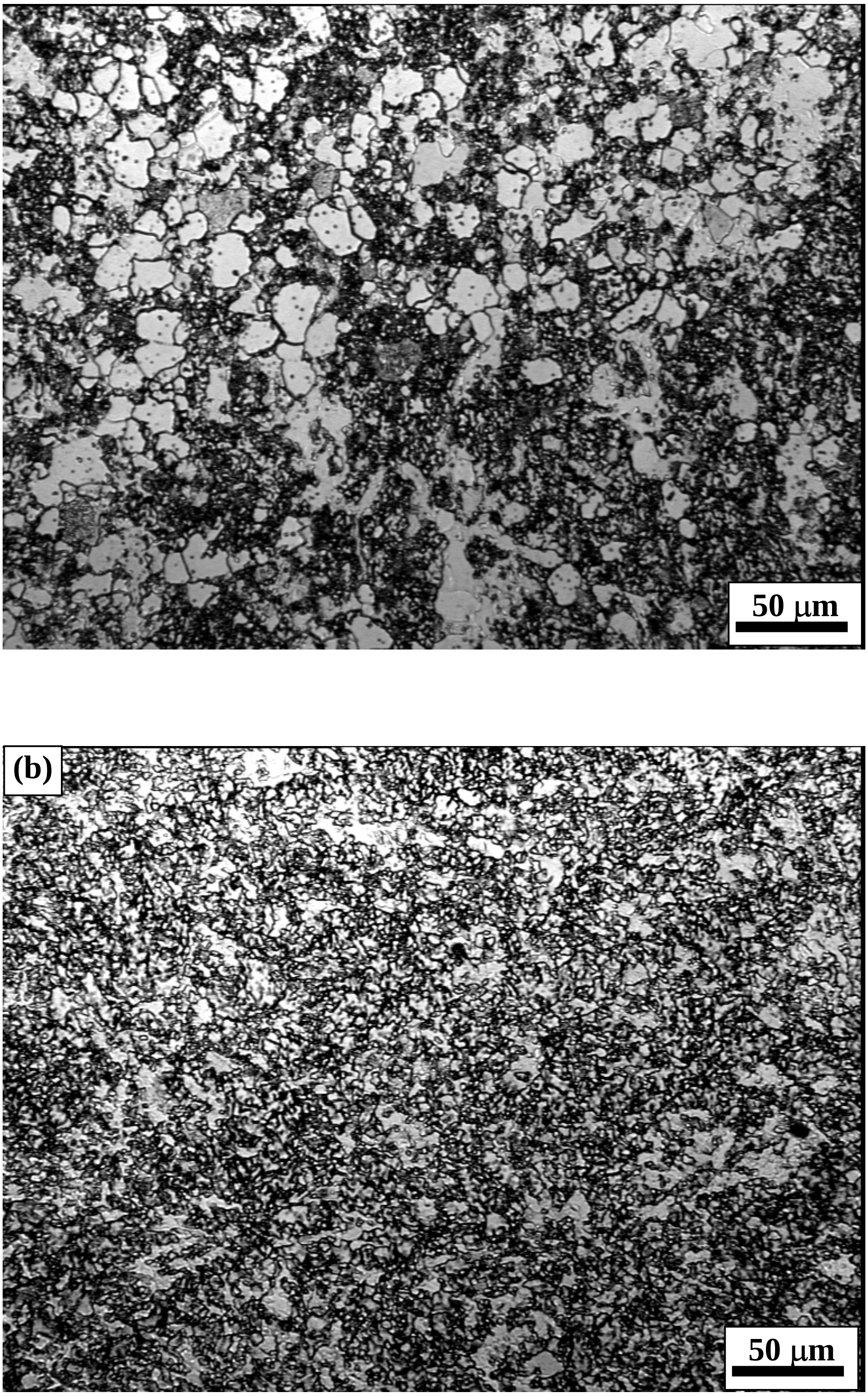


Fig. 9. Microstructure of ECAP W after annealing, 1 hour at a-1150 ^{o}C, b-1000 ^{o}C (cross section)

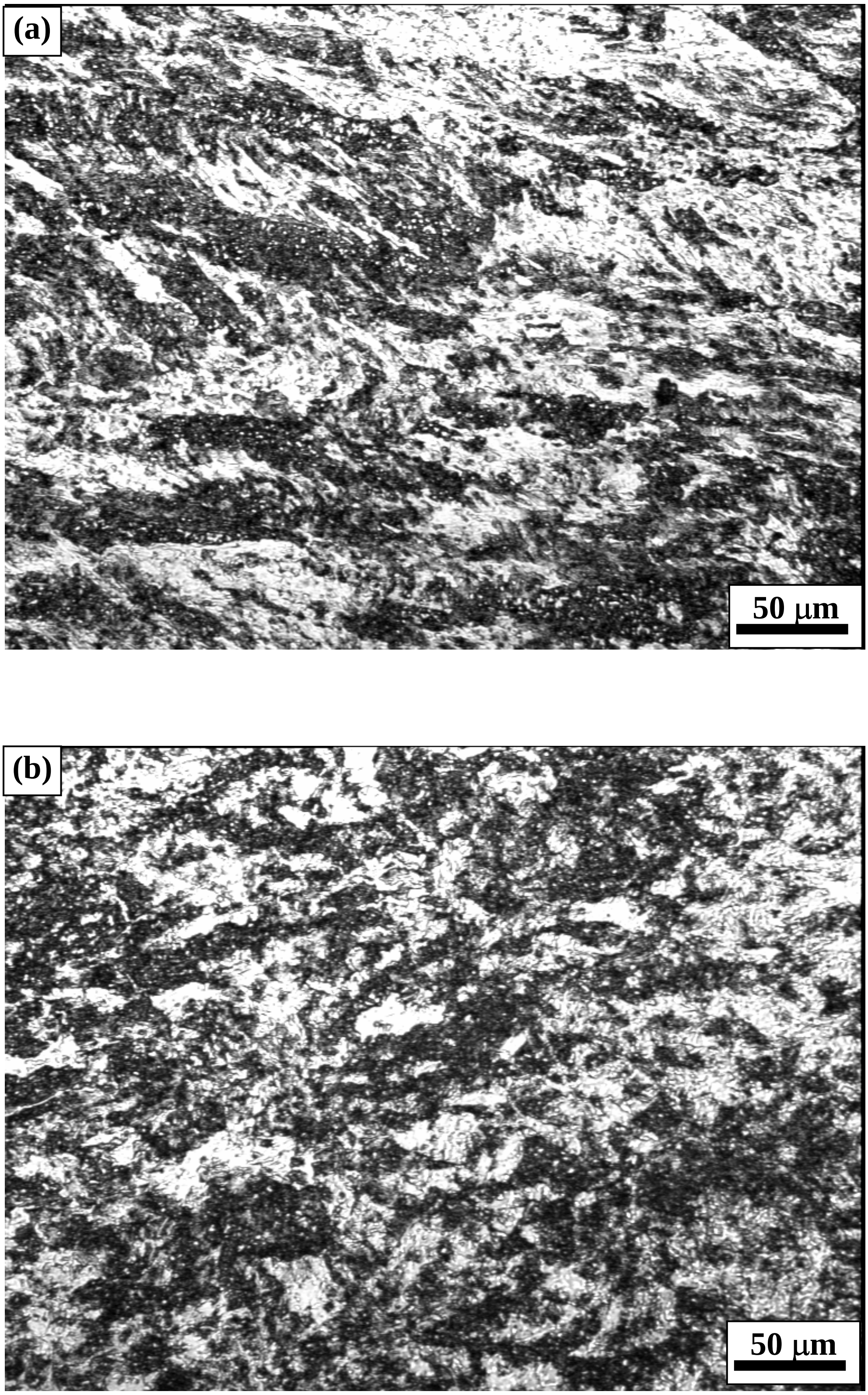
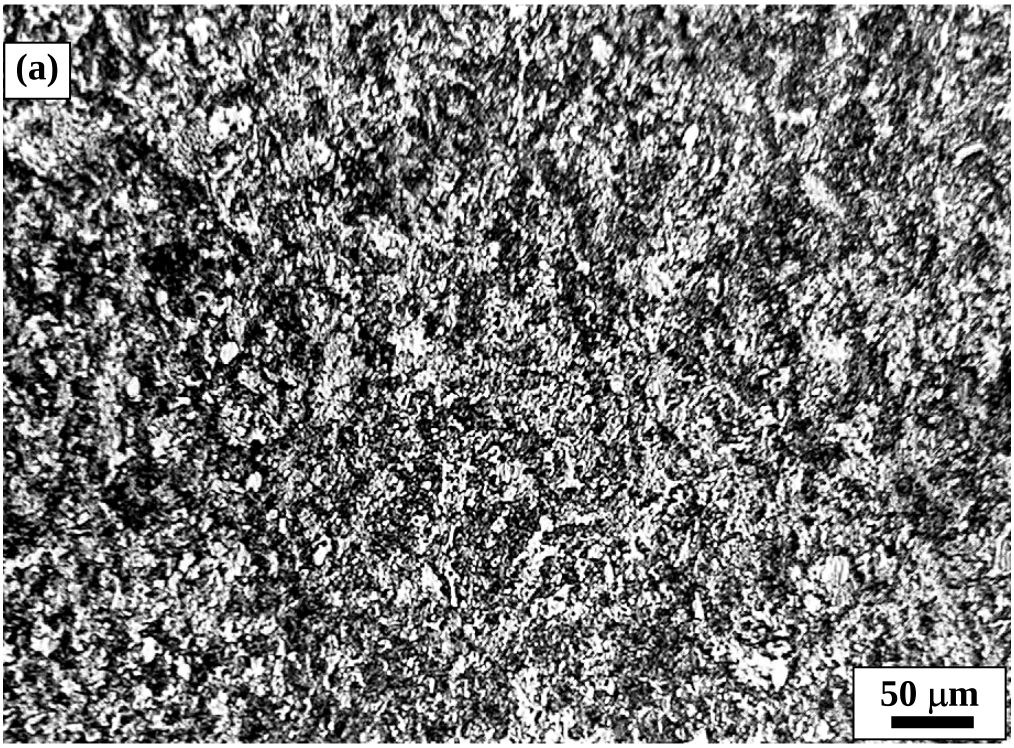


Fig. 10. Metallographic pattern of W after 8 ECAP cycles
at 1150 °C: a- longitudinal section, b- cross section



(a)

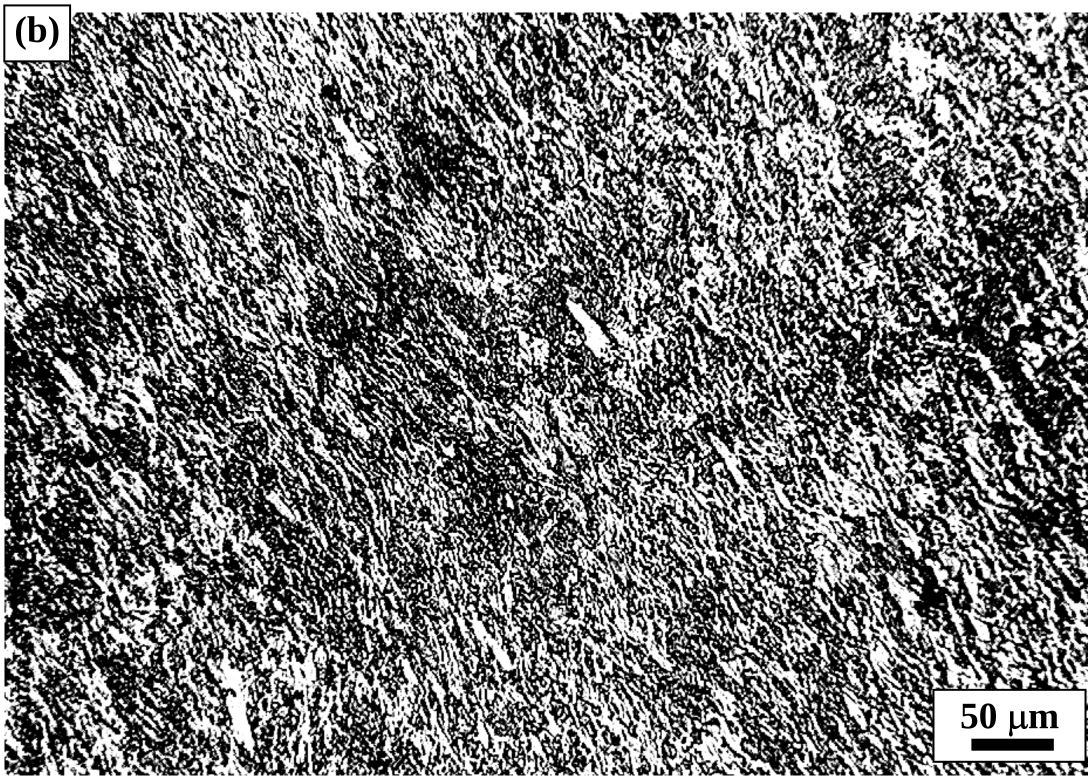


Fig. 11. Metallographic patterns of W after 12 ECAP cycles at 1150 °C: a- cross section, b- longitudinal section

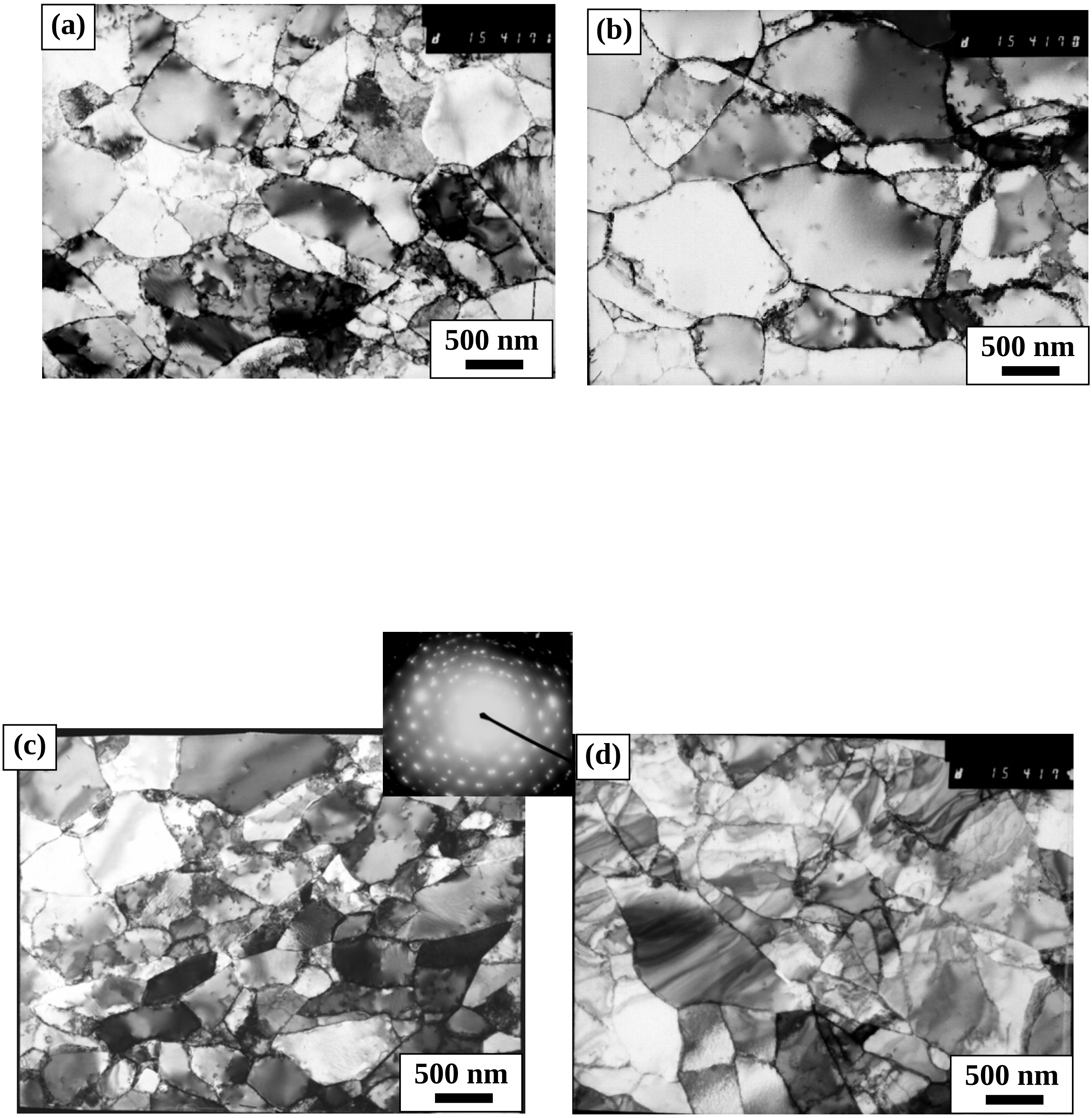


Fig. 12. Electron-microscopic pattern of W dislocation structure after 8 ECAP cycles (a), (b) and 12 cycles (c), (d) at 1150 °C
(a, c- cross section; b, c- longitudinal section)

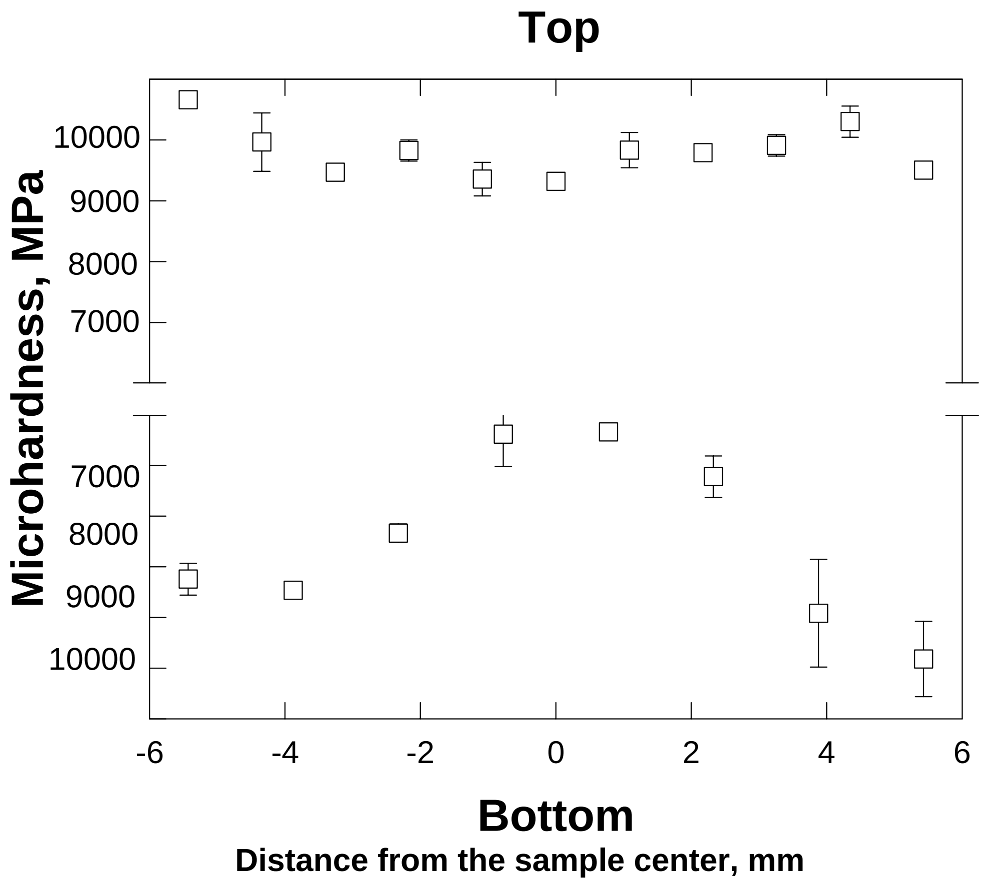


Fig.13. Microhardness of W samples produced by HPT at 500 ^{o}C under pressure of 4.5 GPa

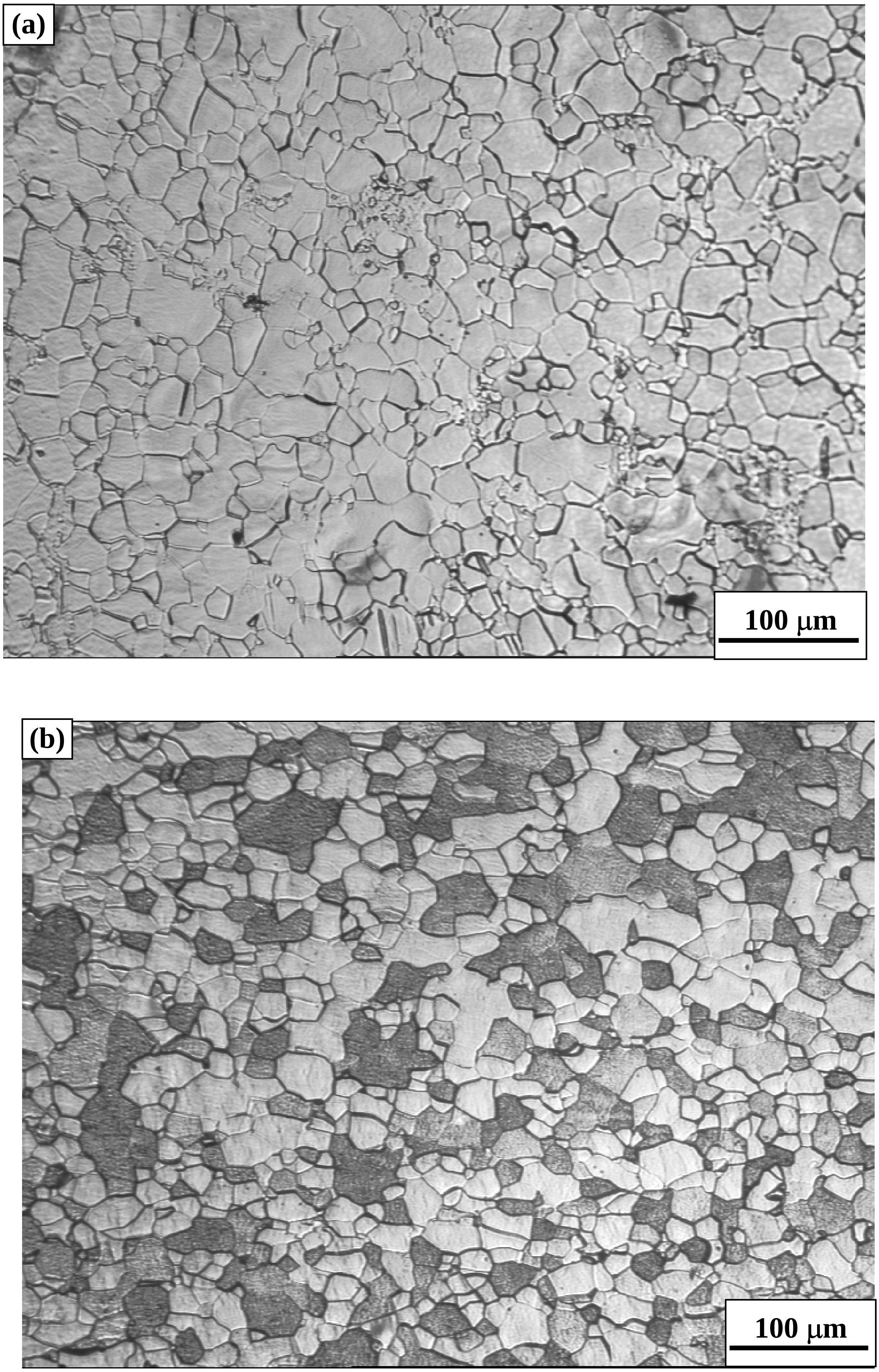


Fig. 14. Metallographic pattern of Ta rod's initial structure
(cross (a) and (b) longitudinal sections)

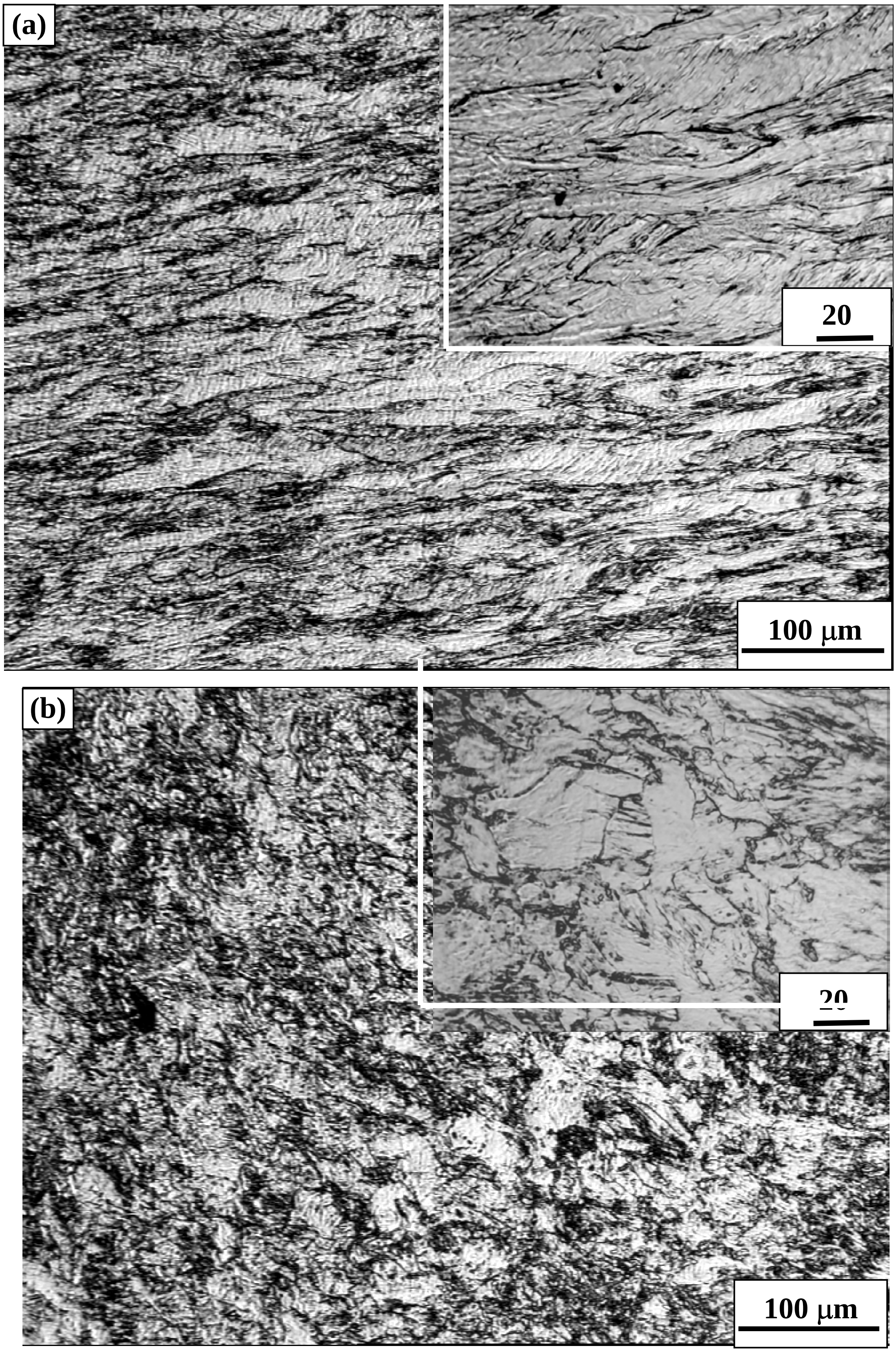
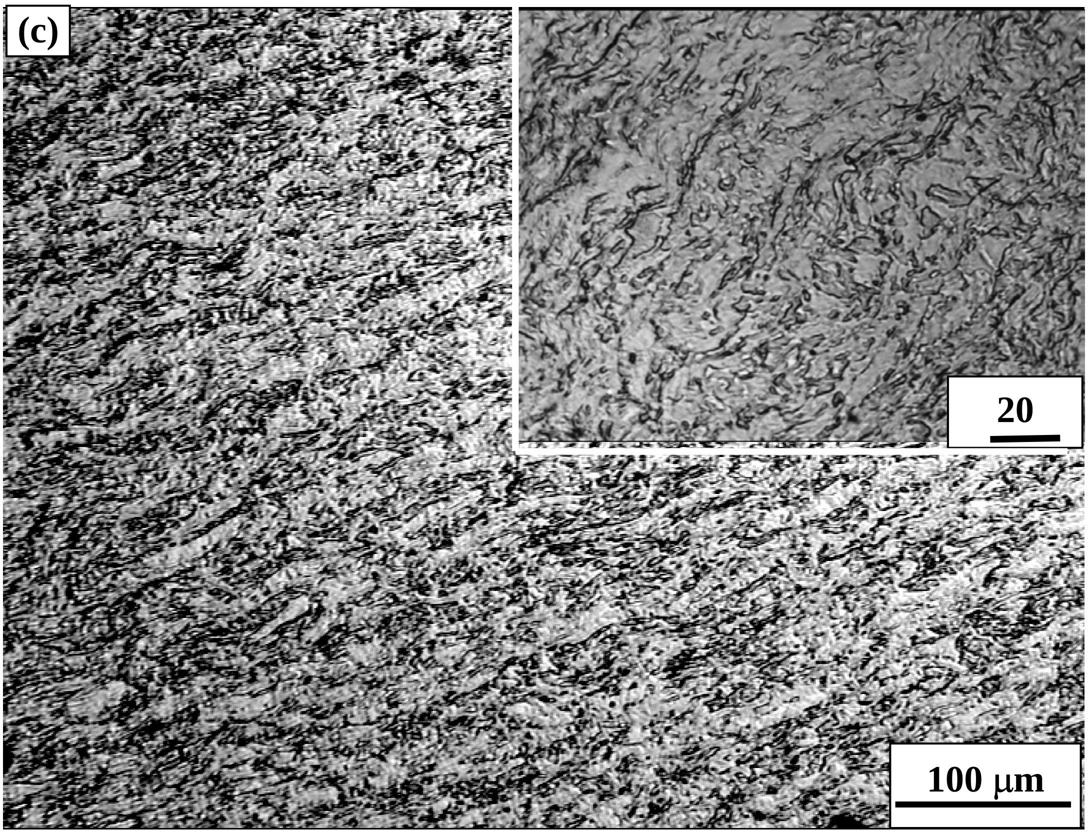


Fig. 15. Metallographic pattern of Ta after 2 cycles of ECAP: a- longitudinal section, b- cross section



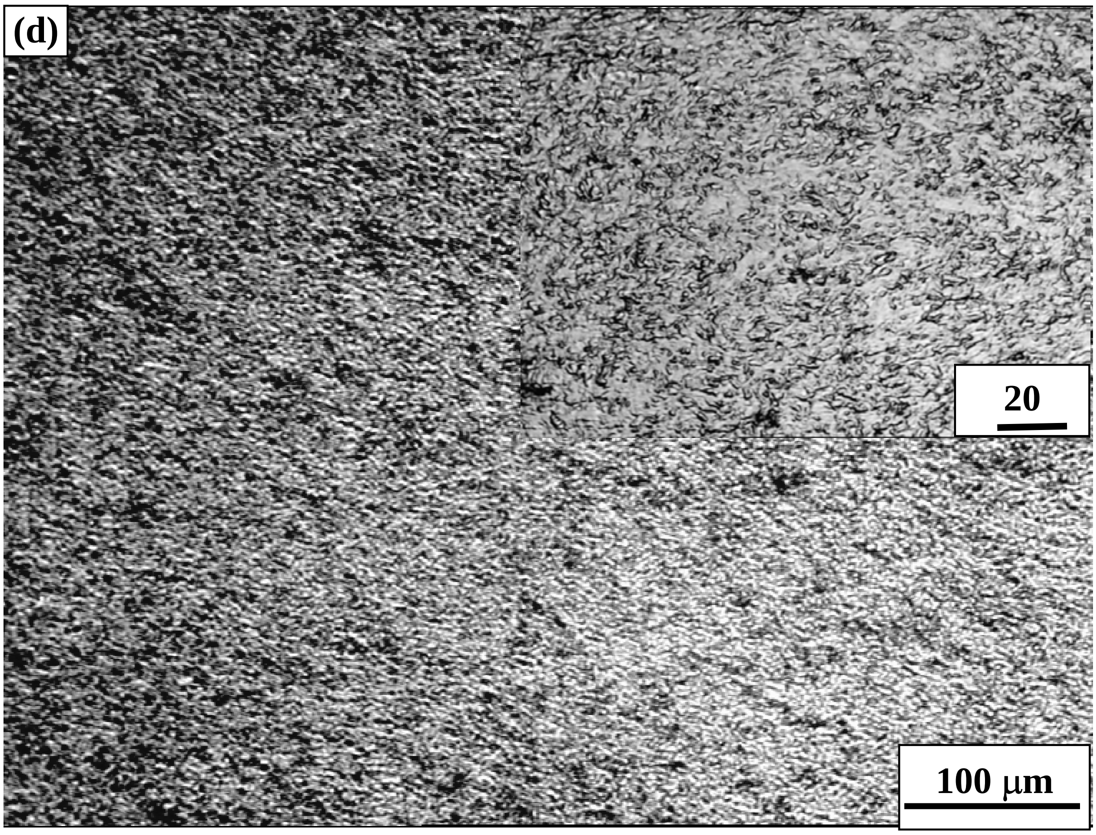
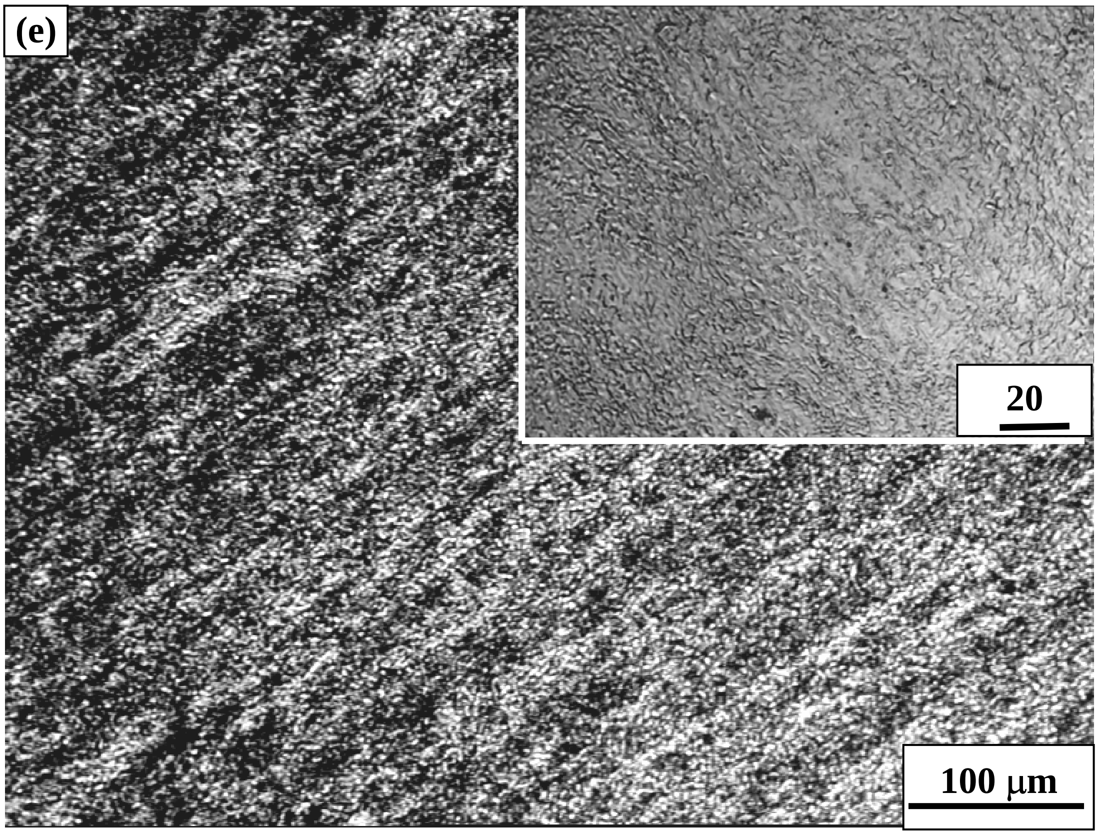


Fig. 15. Metallographic pattern of Ta after 8 cycles of ECAP: c- longitudinal section, d- cross section



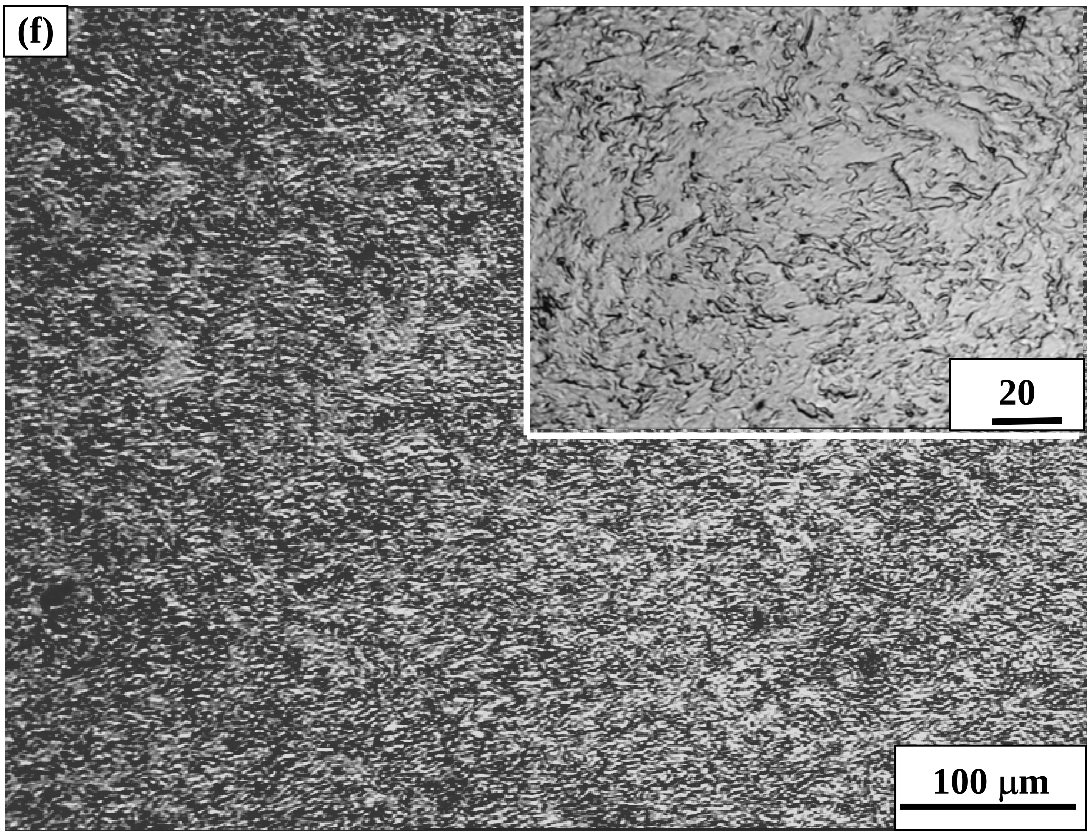


Fig. 15. Metallographic pattern of Ta after 12 cycles of ECAP: e - longitudinal section, f - cross section

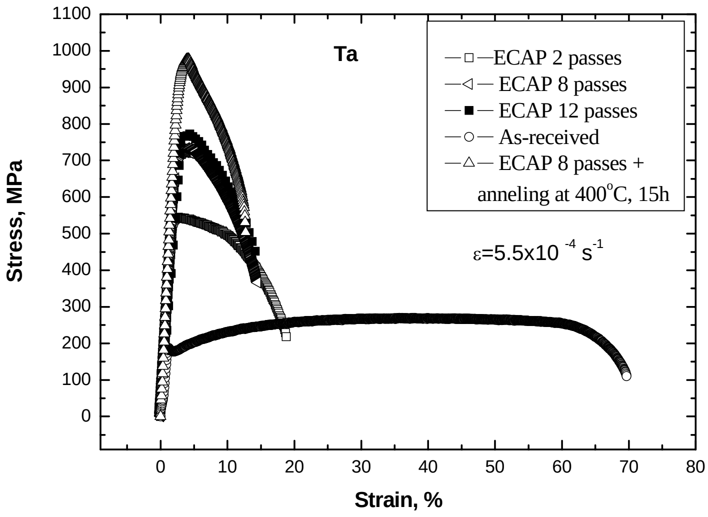


Fig. 16. Engineering tensile curves of Ta before and after ECAP with a different number of cycles

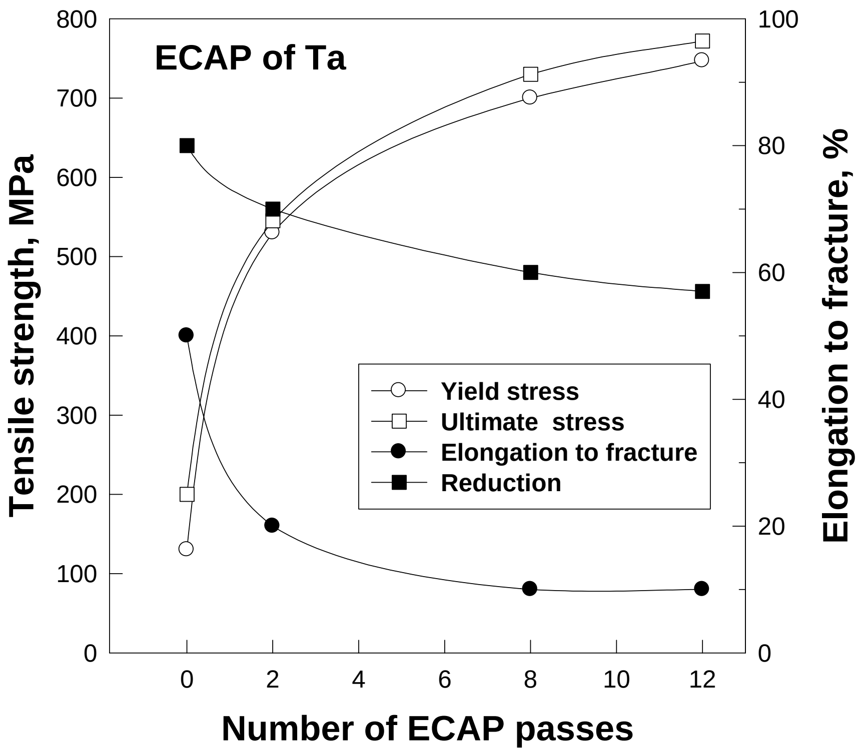


Fig. 17. Dependence of mechanical tensile properties on the number of ECAP cycles of Ta.

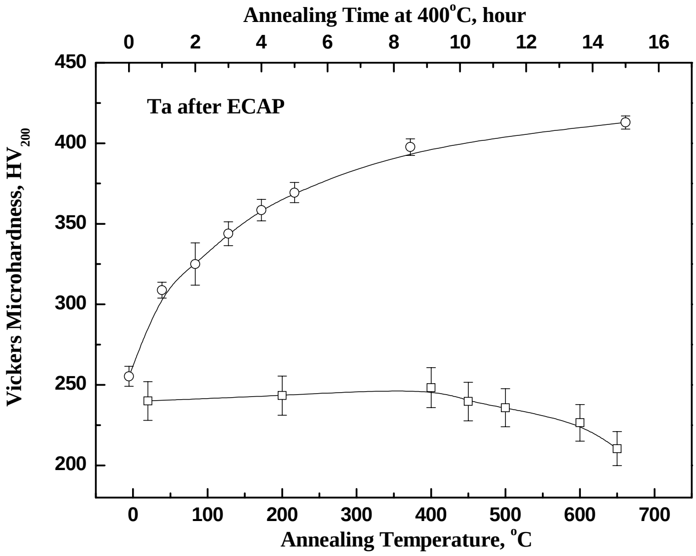


Fig. 18. Dependence of microhardness of ECAP Ta on the temperature and time of heating at 400 °C

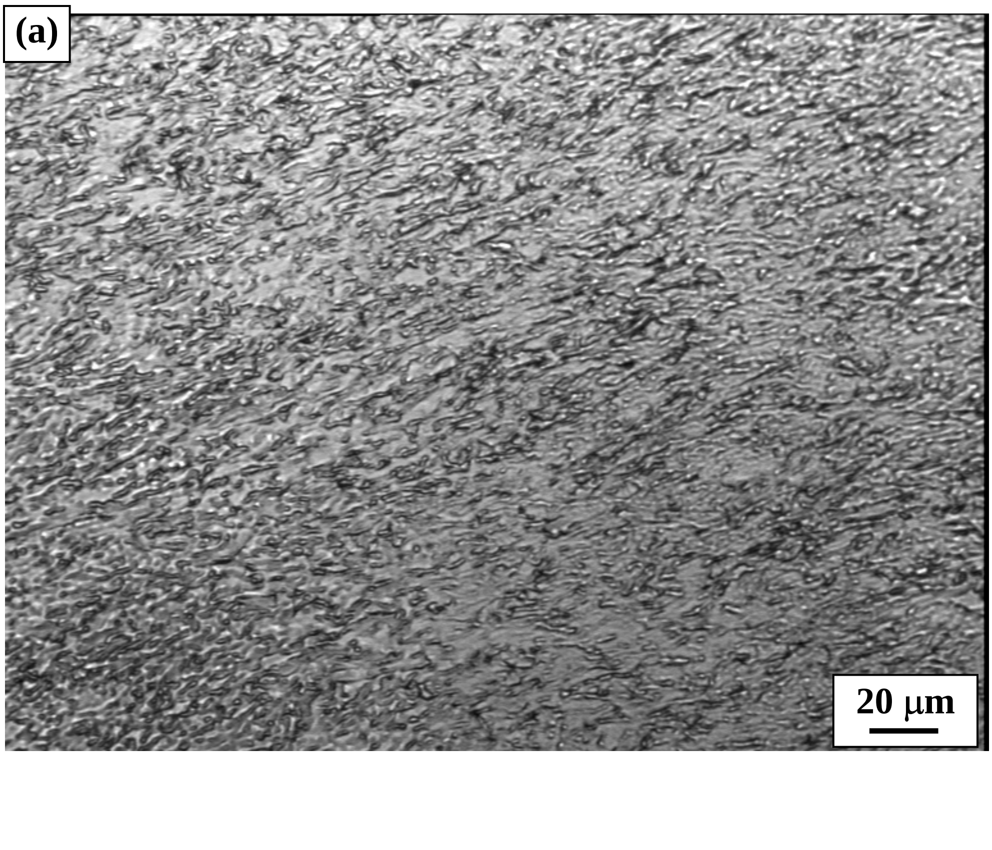


Fig. 19. Metallographic patterns of ECAP Ta after 8 cycles and isotropic annealing at: 400 °C (a), 500 °C (b), 600 °C (c), 650 °C (d)

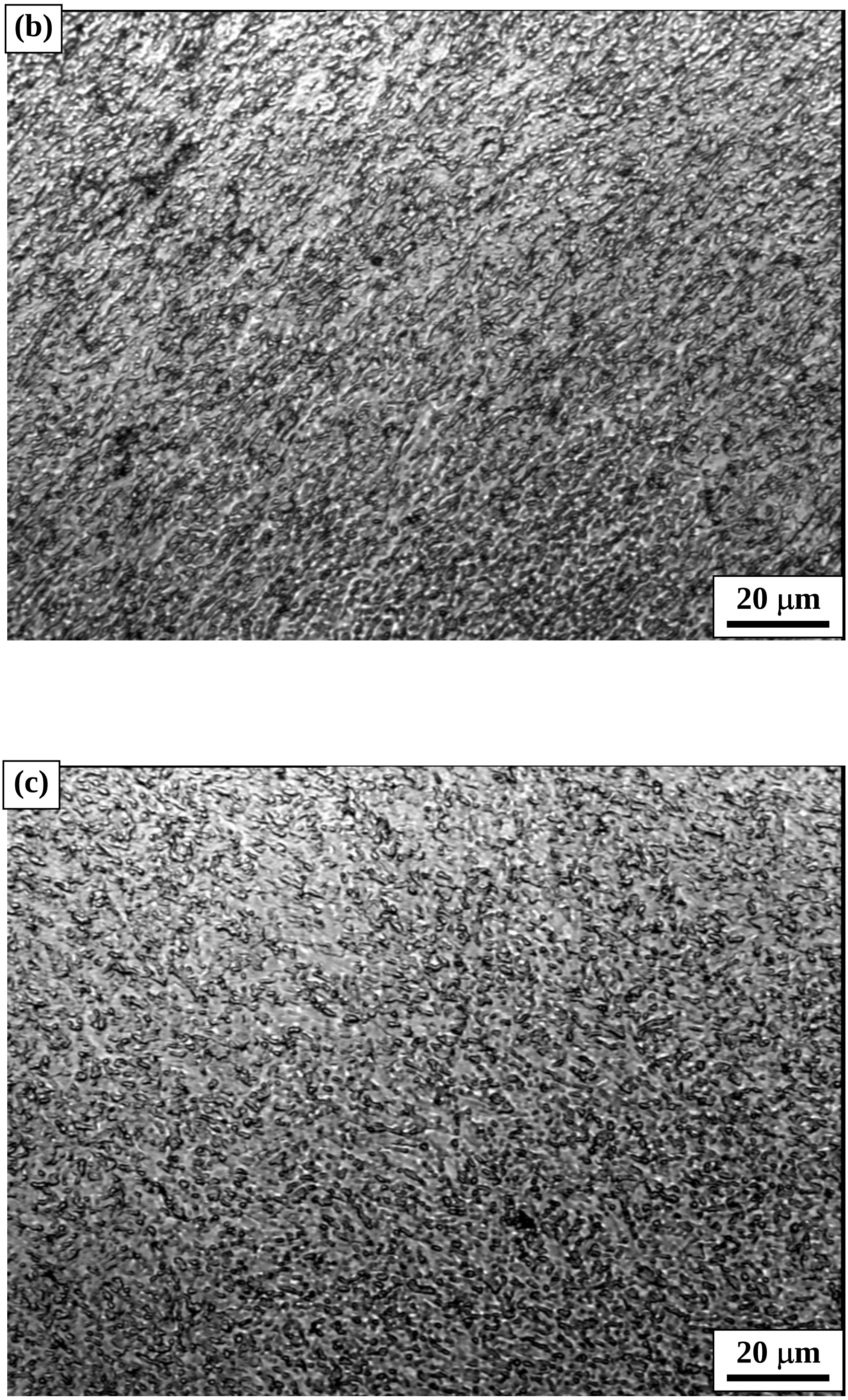


Fig. 19. Metallographic pattern of ECAP Ta structure after 8 cycles and isochronous annealing at: 400 ^{o}C (a), 500 ^{o}C (b), 600 ^{o}C (c), 650 ^{o}C (d)

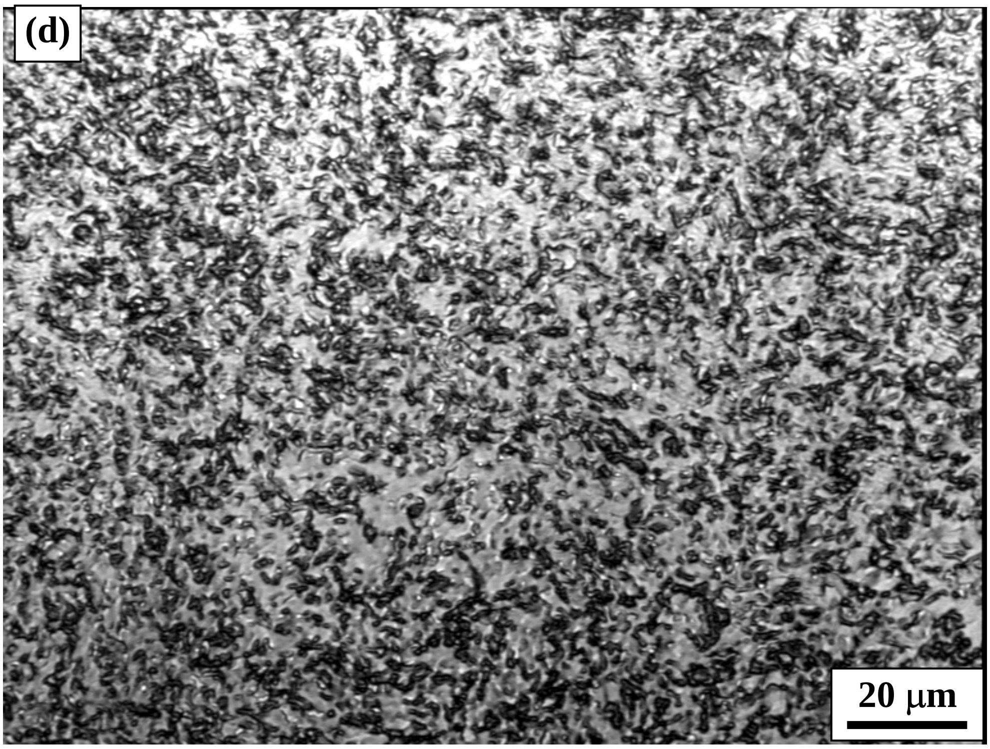


Fig. 19. Metallographic pattern of ECAP Ta structure after 8 cycles and isochronous annealing at: 400 ^{o}C (a), 500 ^{o}C (b), 600 ^{o}C (c), 650 ^{o}C (d)

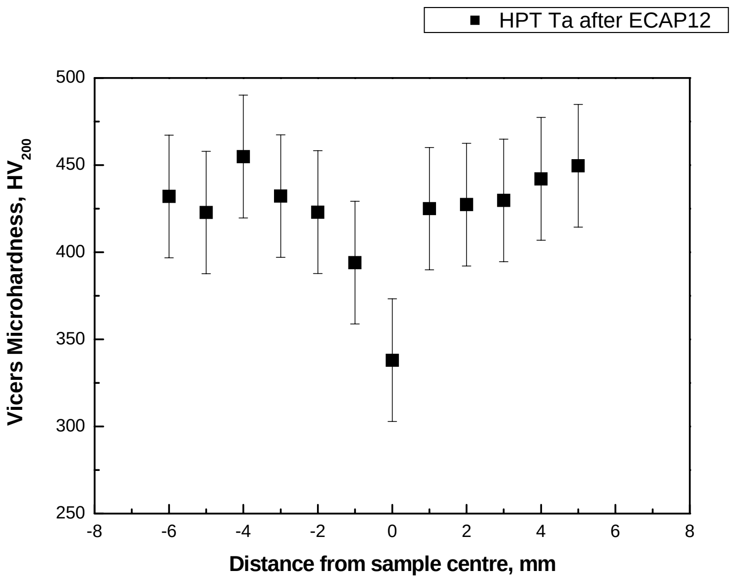


Fig. 20. Distribution of microhardness values along Ta disk's diameter after 12 cycles of ECAP and HPT of 6 GPa +10 turns